



(12) 发明专利申请公开说明书

[21] 申请号 89109675.2

[51] Int.Cl⁵

C07C 53 / 126

[43] 公开日 1991 年 7 月 10 日

[22]申请日 89.12.28

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C07C 51 / 41

说明书页数: 2

附图页数:

[54]发明名称 硬脂酸盐非水一步合成法

[57]摘要

本发明提出了一种硬脂酸的非水一步合成技术。它是以硬脂酸与金属氢氧化物在非水介质中直接合成。本法具有工艺设备简单,生产周期短,效率高,质量好,无氯或硫酸根离子,无可溶性盐,无废液排放。本法可获得显著的经济效益。

(BJ)第1456号

1、一种硬脂酸盐的非水一步合成技术，其特征在于：硬脂酸与金属氢氧化物在非水介质中直接合成，经分离并以有机溶剂和水为洗涤剂，再经干燥、粉碎而得硬脂酸盐。

2、根据权利要求1所述的方法，其特征在于：非水介质为芳烃如甲苯等，其反应温度为50—150℃，用于洗涤的有机溶剂为低级醇如乙醇等。

硬脂酸盐非水一步合成法

本发明提出了一种高质量硬脂酸盐的非水一步合成技术。

硬脂酸盐是有机合成材料中必不可少的一种助剂。例如：硬脂酸钙，它用作聚乙烯、聚丙烯、聚氯乙烯等各种塑料、纤维的稳定剂、润滑剂和卤素吸收剂。对其质量要求很高，有的标准规定了十四项指标，其中：氟化物(以氟离子计)不大于0.01% (从使用上讲，最好无氟)；细度325目通过75%；堆积密度不大于0.2克/厘米³。即要求产品有很高的分散性；熔点 $>150^{\circ}\text{C}$ ；钙含量6.4—7.1%。

硬脂酸盐的生产，历来均采取水相中用氢氧化钠皂化，再与金属可溶性盐复分解反应，生成相应的硬脂酸盐。民主德国专利(241900)发明了一种以金属氧化物或氢氧化物的水分散液与高碳的脂肪酸进行两步或多步反应，制得高分散性，不溶于水的脂肪酸盐的连续合成法。

复分解法需在极稀水液中反应，且洗涤困难(约用100倍的无离子水洗涤)。尽管如此，仍残留少量氟离子或硫酸根离子以及少量可溶性盐。生成品含水量很高，干燥费时，同时还有大量含盐废水排放。总之，该法最大缺点是

生产效率很低，且质量也不稳定。前述专利优点是：产品中无氯或硫酸根离子，不需大量水洗涤，无含盐废水。但该工艺仍属水相反应，产品产量和质量，还受到一定限制。

本发明以芳烃为反应介质，硬脂酸与金属氢氧化物直接一步合成硬脂酸盐，再以低级醇和水洗涤，经分离、干燥、粉碎而得成品。

一个硬脂酸钙的合成实例是：按比例称取硬脂酸和氢氧化钙（硬脂酸适当过量），加入反应器中，再加入甲苯，其液固比为：1—20:1 (V/W)，搅拌升温，在50—150℃反应1—4小时，冷至室温，分离出固体，再以乙醇和水洗涤，最后经干燥、粉碎而得。产品经测定：堆积密度为0.16克/厘米³，无氯无可溶性盐，熔点155℃，钙含量6.1%。

可以看出，本法具有如下优点：

- 1、工艺设备简单，生产周期短、效率高、质量好。
- 2、无氯或硫酸根离子，无其他可溶性盐。
- 3、不用大量纯水。
- 4、无废水排放，溶剂可回收使用。

One-step nonaqueous synthesizing process of stearate

The present invention relates to a method for nonaqueous synthesis of high-quality stearate only by one step.

Stearate is an indispensable adjuvant in organic synthetic material. For example, calcium stearate, it can be used as stabilizer, lubricant and halogen absorber for various plastics such as polyethylene, polypropylene and polyvinyl chloride, and fibers. There is a high requirement on its quality, and fourteen indexes are specified therefor in some standard, including chloride (calculated as chlorine ions): not more than 0.01% (in use, chlorine-free is the most preferred), fineness: 75% of the product passing 325 meshes, stacking density: not more than 0.2 g/cm³, i.e., the product having a high dispersibility, and melting point: $\geq 150^{\circ}\text{C}$, and calcium content: 6.4-7.0%.

The production of stearate has been always carried out by saponifying with sodium hydroxide in aqueous phase, followed by double decomposition reaction with a metal soluble salt, to thereby obtain a corresponding stearate. The Germany patent (241900) discloses a continuous synthetic process comprising subjecting an aqueous dispersion of a metal oxide or hydroxide and a higher fatty acid to two or multiple-step reaction, to thereby obtain a high-dispersible, water insoluble fatty acid salt.

Double decomposition process shall be carried out in a very dilute aqueous solution, and it is difficult to do washing (about 100 times of deionized water is required for washing). Even so, there still remains a few amount of chlorine or sulphuric radical ions and a few amount of soluble salt in the product. The product contains a large quantity of water, so it takes a lot of time for drying, and meanwhile a large quantity of salt-containing waste liquid is discharged. To sum up, the maximum demerits of this process reside in very low production efficiency and instable quality. The merits of the aforesaid patent reside in: the product is free of chlorine or sulphuric radical ions, do not need a large quantity of water for washing, and no salt-containing waste liquid is discharged.

However, this process still belongs to aqueous phase reaction, and the output and quality of the product are still restricted in certain extent.

The present invention relates to reacting stearic acid and a metal hydroxide in an aromatic hydrocarbon as reaction medium for one-step synthesis of a stearate, washing it with a lower alcohol and water, separating, drying, and crushing to obtain the product.

One example of synthesizing calcium stearate is described as follows: stearic acid and calcium hydroxide (an appropriate excess of stearic acid) were weighted in a certain ratio, and added in a reactor, to which toluene was further added, resulting in a liquid/solid ratio of 4-20:1 (v/w); the reaction mixture was heated with stirring, reacted at 50-150°C for 1-3 h, and then cooled down to room temperature; thereafter, solids were separated, washed with ethanol and water, and finally dried and crushed to obtain the product. As determined, the product had the following properties: stacking density 0.16 g/cm³, free of chlorine and soluble salt, melting point 155°C, and calcium content 6.8%.

It can be seen that the process of this invention has the following merits:

1. Simple technology and apparatus, short producing cycle, high efficiency and quality;
2. Free of chlorine or sulphuric radical ions, and free of other soluble salt;
3. No use of a large quantity of pure water;
4. No waste liquid discharging, and reusable solvent.

Claims

1. One-step nonaqueous synthesizing process of stearate, comprising reacting stearic acid and metal hydroxide in a nonaqueous medium for the direct synthesis of a stearate, separating and washing it with an organic solvent and water, and then drying and crushing to obtain the product.
2. The process according to claim 1, wherein the nonaqueous medium is an aromatic hydrocarbon such as toluene, the reaction temperature is 50-150°C,

and the organic solvent for washing is a lower alcohol such as ethanol.

Abstract

The present invention relates to one-step nonaqueous synthesizing process of stearate, wherein stearic acid and metal hydroxide are used for the direct synthesis in a nonaqueous medium. The process is featured with simple technology and apparatus, short producing cycle, high efficiency and quality; free of chlorine or sulphuric radical ions, and free of other soluble salt; and no waste liquid discharging.